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Indian Standard

METHODS OF SAMPLING AND TEST FOR PRESSURE SENSITIVE ADHESIVE TAPES

(First Revision)

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Gr 3

May 1988

Indian Standard

METHODS OF SAMPLING AND TEST FOR PRESSURE SENSITIVE ADHESIVE TAPES

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 10 December 1987, after the draft finalized by the Adhesives Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Conncil.

0.2 Pressure sensitive adhesive tapes are generally used for sealing the containers, providing a means of reinforcement when necessary and to protect the contents from outside contamination or damage and in the case of waterproof tapes, also retard the entry of water and transmission of water vapour. These tapes need only the application of gentle pressure to cause them to adhere to almost any clean dry surface which is practically free from dust, oil, grease or moisture.

0.3 In the original version, this standard covered pressure sensitive tapes, such as, cellulose, cloth and PVC tapes. The Committee took cognizance of the usage of newer plastic substrates other than PVC like polypropylene, polyethylene and others and examined the relevance of these test methods to adhesive tapes made from plastics other than PVC and has concluded that the test methods given in this revised version would suffice these exigencies as well.

0.4 This standard was first published in 1977. Based on the experience gained through the use of this standard, the Committee felt that the following three tests which were earlier prescribed in the product specification as optional are redundant and, have therefore, been deleted in this revision:

i) *p*H,

ii) Chloride content, and

iii) Sulphate content.

0.4.1 At one stage, the Committee felt that stability test and durability test were partly duplicating each other and, therefore, a modified durability test to cover stability also was incorporated in the revision of product specifications (*see* IS : 2880-1986[*] and IS : 3676-1986[†]). Later on, it was felt that while stability test indicates adhesive behaviour, the durability test is relevant to application characteristics. In view of this, the Committee decided to retain stability test along with the modified durability test in this revision.

0.4.2 Some individual product standards for tapes included two tests, namely, unwind characteristics and adhesion to self. Since unwind characteristics test was partly duplicating adhesion to self test, the later has been suitably amplified and generalized in this revision to cover the assessment under unwind characteristic test also. Since unwind test was not a part of this standard, it is not being included. As a consequence, unwind test would be deleted from relevant product specifications (namely IS : 2880-1986[*] and IS : 3676-1986[†]).

0.5 This revised methods of test standard accordingly covers rationalized performance tests which are expected to be essential for a quick performance appraisal of adhesive tape within minimum testing time.

0.6 The relative importance of the tests described depends on the purpose for which the adhesive tape is intended. In practice, it is customary for the user to determine by experience the most suitable type of adhesive tape for this purpose. The problem then reduces itself to checking future deliveries in respect of those properties which are relevant. If these methods are carefully followed in every detail, it is possible to obtain consistent results which will give useful information to the manufacturer and the user.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960[‡]. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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[*] Specification for pressure sensitive adhesive cellulose tapes (*third revision*).

[†] Specification for pressure sensitive adhesive PVC tapes (*second revision*).

[*] Specification for pressure sensitive adhesive cellulose tapes (*third revision*).

[†] Specification for pressure sensitive adhesive PVC tapes (*second revision*).

[‡] Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the methods of sampling and test for pressure sensitive adhesive tapes, such as cloth tapes, PVC tapes, cellulose tapes and a variety of plastic film/base materials.

1.2 This standard is not intended to apply to rubber latex or to adhesives used for miscellaneous purposes or those manufactured from materials of vegetable origin.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 3434-1984* shall apply.

3. SAMPLING

3.1 Lot — In any consignment, the rolls of tape made from the same backing material and one lot of adhesive and manufactured under the same conditions shall constitute a lot.

3.1.1 Tests for determining the conformity of the lot to the requirements of the specification shall be done on each lot separately. The number of rolls to be selected from a lot shall depend upon the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

TABLE 1 NUMBER OF ROLLS TO BE SELECTED FOR SAMPLING

LOT SIZE *N* (1)	NO. OF ROLLS TO BE SELECTED *n* (2)
Up to 100	2
101 " 500	5
501 " 1 000	7
1 001 and above	10

3.1.2 The rolls shall be selected at random and in order to ensure the randomness of selection, a random number table (*see* IS : 4905-1968†) shall be used. In case such a table is not available, the following procedure may be adopted:

Starting from any roll, count them in one order as 1, 2, 3, ... , up to *r* and so on, where *r* is the integral part of *N*/*n* (*N* being the lot size and *n* the number of rolls to be selected). Every *r*th roll thus counted shall be withdrawn to give samples for test.

4. GENERAL DIRECTIONS

4.1 Conditioning — Each roll selected for test shall be conditioned for a minimum period of 24 hours at 27 ± 2°C and 65 ± 5 percent relative humidity (*see* IS : 196-1966‡) prior to testing, and testing shall be preferably done in the same atmosphere. Where the tests cannot be carried out in the same atmosphere, the testing shall be commenced within two minutes of withdrawal of the specimens from the conditioning atmosphere.

4.2 The outer three layers of each roll shall be discarded before taking the specimens for test.

4.3 All specimens shall be removed from the roll at an approximate speed of 30 cm/min.

4.4 The adhesive surface shall not be permitted to come in contact with fingers, to be contaminated with dust, or to come in contact with other foreign matter.

4.5 Quality of Reagents — Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of test.

5. DETERMINATION OF ADHESION STRENGTH TO METAL

5.0 Outline of the Method — Adhesion strength is determined by finding the force required to peel a strip of tape from a standard test panel at a specified angle and speed.

5.1 Number of Tests — Carry out the determination on five test specimens taken from the same roll.

5.2 Apparatus

5.2.1 *Tensile Testing Machine* — A pendulum or spring balance type tensile testing machine, with a uniform rate of travel of 30 cm/min of the moving head or carriage, and a scale graduated to 0·05 kg or less.

5.2.2 *Stainless Steel Plates* — Rectangular, 10 × 30 cm polished stainless steel plates (*see* Grade 07Cr18Ni9 of IS : 6911-1972†).

5.2.2.1 The stainless steel plates shall be finished in the lengthwise direction to a surface roughness not greater than 80 µm (*see* IS : 3073-1967‡).

5.2.2.2 The stainless steel plates shall also be marked boldly or etched at intervals of 2 cm along both the longitudinal edges.

5.2.3 A steel roller of 80 ± 2 mm diameter and 50 ± 1 mm in width covered with rubber approximately 6 mm thick having hardness of 80 ± 5 IRHD. The mass of the roller which applies pressure to that specimen shall be 2·05 ± 0·05 kg. It shall be so constructed that the mass of the handle is not added to the mass of the roller during use (*see* Fig. 1).

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*Glossary of terms for adhesives and pressure sensitive adhesive tapes (*first revision*).

†Methods for random sampling.

‡Atmospheric conditions for testing (*revised*).

*Specification for water for general laboratory use (*second revision*).

†Specification for stainless steel plate, sheet and strip.

‡Assessment of surface roughness.

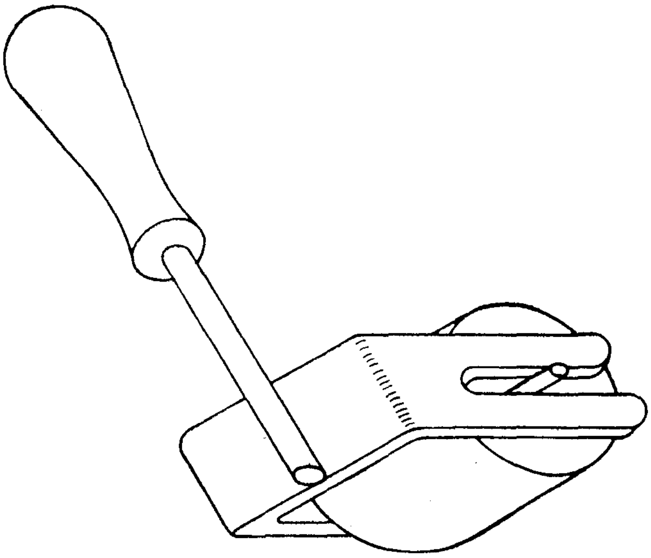

FIG. 1 ROLLER FOR ADHESION STRENGTH TEST

5.3 Preparation of Test Specimens — Condition the rolls as specified in **4.1** and take all precautions as specified in **4.2** to **4.4** for taking the test specimen. Test specimens shall be 0·5 to 3 cm in width and not less than 50 cm in length. If the width of tape is greater than 3 cm, then reduce it to 3 cm by cutting with a sharp blade.

5.4 Procedure

5.4.1 *Preparation of Steel Plate* — Clean thoroughly the surface of the stainless steel plate with re-distilled toluene using a clean piece of untreated tissue paper or cotton wool or cotton cloth for each cleaning. Suspend the plate in the toluene vapour bath for 5 minutes after the vapour line has reached the top of the plate, so that no liquid is in contact with the plate. Allow the plate to cool for approximately 30 minutes in an atmosphere maintained at a temperature of 27 ± 2°C and 65 ± 5 percent relative humidity so that the plate attains a temperature of 27 ± 2°C.

5.4.2 *Application of Test Specimen* — Place the cleaned plate, test surface upwards, on the table. Apply at least 25 cm of the test specimen without stretching, adhesive side down, to the plate so that the tape lies centrally on the plate and parallel to the longer side leaving the remainder of the test specimen extending beyond the steel plate sufficient to be accommodated in the testing equipment. Precautions shall be taken so that no air bubbles are trapped between the tape and the plate.

NOTE — In the case of tapes less than 3 cm wide, cut other strips from the same sample roll and apply them parallel and adjacent to the test specimen to provide a total width of 3 cm for rolling purposes only.

5.4.2.1 Place the roller centrally across the test specimen at one end of the plate and pass the roller once in each direction at constant speed of approximately 30 cm/min, ensuring that no additional pressure on the roller is applied during the process. Allow the steel plate with the test specimen to remain undisturbed for 10 ± 0·5 minutes at a temperature of 27 ± 2°C and 65 ± 5 percent relative humidity.

5.4.3 Fold the free end of the test specimen at an angle of 180° and peel off 3 cm from the steel plate leaving at least 22 cm in contact with the steel plate. Clamp this exposed end of the plate in the lower jaw (while using a vertical pendulum or spring balance type machine), or attach the whole plate to the moving carriage (if the testing machine is of the horizontal type). Attach the free end of the tape to the head of the tension measuring device and disengage the pawls, if any. Start the tensile testing machine and take readings at 2 cm intervals when the tape is pulled from the steel plate, disregarding the pulling of first 5 cm and last 3 cm. The mean of five readings shall give the results for individual test specimens, that is, the load required to cause the separation of the tape from the steel plate. If the specimen breaks during the test, repeat the test on another specimen cut from the same roll.

5.5 Calculation and Reporting — Calculate the load required to cause the separation of the tape from the steel plate in terms of kg/cm width. If the variation of any of the five results exceeds ±10 percent of the mean of the five results, all of them shall be discarded and a further set of five test specimens shall be tested. The mean of the fresh five values obtained shall be reported as adhesion strength to the metal.

6. DETERMINATION OF BREAKING STRENGTH AND ELONGATION AT BREAK

6.0 Outline of the Method — Breaking strength is determined by finding the load at which the tape breaks, when tested on a tensile testing machine and elongation is determined by finding the increase in length from the original length at the breaking point.

6.1 Number of Tests — Carry out the determination on three test specimens taken from the same roll.

6.2 Apparatus

6.2.1 *Tensile Testing Machine* — A pendulum type tensile testing machine with a uniform rate of jaw separation of 30 cm/min and a scale graduated to 0·2 kg or less.

6.3 Conditioning — Condition the rolls as specified in **4.1** and take all precautions as specified in **4.2** to **4.4** for taking the test specimens.

6.4 Procedure — Take test specimens of sufficient length to accommodate in a jaw separation of 12 cm and measure its width (*see* Note). Clamp the strip of tape squarely in the jaw of the tensile testing machine and start the machine. Note the load and elongation at break. Reject the test pieces which break at or in the jaws and repeat the test.

NOTE — The width of tape shall not vary by more than ±1 mm from the specified width and shall be checked by taking five determinations at random in a length of not less than 1 metre of tape from each sample roll.

6.5 Calculation and Reporting

6.5.1 *Breaking Strength* — The mean of three determinations shall be taken as the breaking strength in N per 10 mm width or kgf per 10 mm width.

6.5.2 *Elongation at Break* — Calculate the percentage, elongation by dividing the extension by original distance between the jaws and multiplying by 100. Report the mean of three determinations as elongation at break.

7. TEST FOR ANCHORAGE

7.0 Outline of the Method — Anchorage is tested by observing the adhesive mass transfer from one face to another when the adhesive sides of the tape are joined together face to face under a specified pressure.

7.1 Procedure — Condition the rolls as prescribed in **4.1** and take 30 cm strip of tape from the roll while taking precautions given in **4.2** to **4.4**. Fold the strip squarely so that adhesive side of the folded strip is face to face. Pass the roller as given in **5.2.3** at a rate of approximately 5 cm/min. Unfold the strip at a rate of approximately 5 cm/min and observe the adhesive side for anchorage.

8. TEST FOR FREEDOM FROM DELETERIOUS EFFECT ON PLASTICS SHEET

8.0 Outline of the Method — Deleterious effect on plastics sheet is determined by applying tape on a plastic surface and examining the tape for signs of transferred adhesive mass.

8.1 Apparatus

8.1.1 *Plastics Strip* — of clear unplasticized methyl methacrylate, 150 mm long and 25 mm wide, cut from a flat 3 mm thick sheet of cast material. Use a new strip for each test.

8.1.2 *Mandrel* — of 75 cm radius.

8.2 Procedure — Remove the protective paper from the plastics strip and free the strip from any residual adhesive by soaking in distilled water at room temperature for approximately 30 minutes. Rinse the strip with distilled water, allow it to dry in air at room temperature and lightly wipe it with a clean dry polishing cloth. Stress the strip by clamping it lightly at each end so that it is bent flatwise round the mandrel (*see* Fig. 2). Place a piece of the adhesive tape, approximately 6 cm long, centrally on the strip and roll it to ensure close contact between adhesive mass and the outer surface of the strip. Store the assembly for 24 hours in an atmosphere free from vapours of organic solvents, at a temperature of 20 ± 2°C. At the end of that period, pull the tape radially from the strip and examine the strip visually for signs of transferred adhesive and by transmitted light at varying angles of incidence for crazing.

9. TEST FOR RESISTANCE TO PENETRATION BY PAINT SOLVENTS

9.0 Outline of the Method — Resistance to penetration by paint solvents is determined by applying tape to a plastic surface in presence of a drop of paint solvent and examining the tape for transfer of adhesive mass.

9.1 Apparatus

9.1.1 *Plastics Strip* — Same as in **8.1.1**. Use a new strip for each test.

9.1.2 *Mandrel* — Same as in **8.1.2**.

9.2 Reagents

9.2.1 *Solvent A* — Prepared by mixing butyl acetate, toluene, amyl acetate and butyl alcohol in the ratio of 4 : 2·5 : 1 : 2·5.

9.2.2 *Solvent B* — Prepared by mixing xylene and butyl alcohol in the ratio of 9 : 1.

9.3 Procedure

9.3.1 Apply a piece of tape to the strip of plastics sheet which has been cleaned and stressed round a mandrel in exactly the same manner as in **8.2.** Place a drop, approximately 0·02 ml, of solvent A centrally on the upper surface of the tape, taking care to prevent the liquid from reaching the edges. Store the assembly for two hours in draught-free atmosphere at 20 ± 2°C. At the end of that period, pull the tape radially from the strip and examine the strip visually for signs of transferred adhesive and by transmitted light at varying angles of incidence for crazing.

9.3.2 Make a similar test using a drop, approximately 0·02 ml, of solvent B.

10. DETERMINATION OF ADHESION AFTER IMMERSION IN WATER

10.0 Outline of the Method — Adhesion to metal and self is determined after immersing the tape in water for 24 hours.

10.1 Procedure — Prepare five test specimens each for adhesion to metal and self by the method prescribed in **5.4** and **13.1**. Immerse these specimens in water at 27 ± 2°C for 24 hours. Test immediately after removing the surplus water with a clean absorbent cloth or paper, for adhesion to metal and self as prescribed in **5** and **13**.

10.2 Calculation and Reporting — Calculate the load required to cause the separation of tape from the steel plate and self in terms of kilograms per

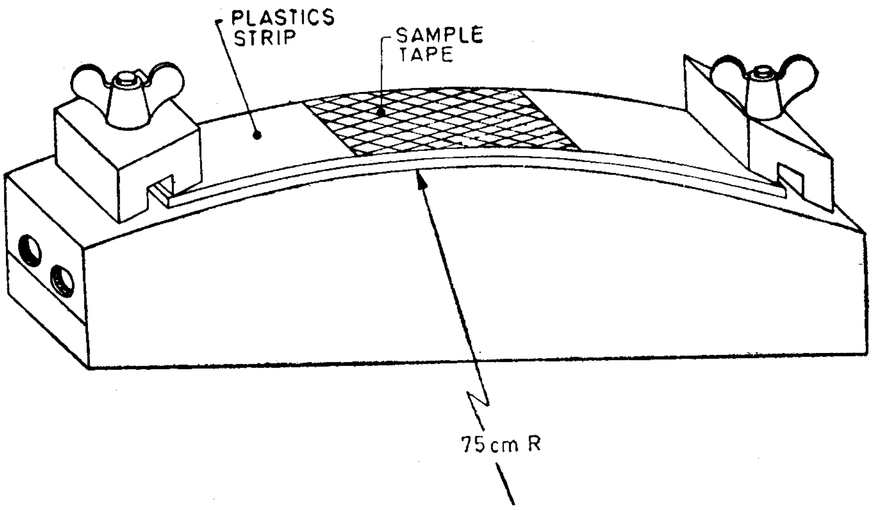

FIG. 2 MANDREL

centimetre width. If the variation of any of the five results exceeds ±10 percent of the mean of the five test results, the results shall be discarded and a further set of five test specimens shall be tested. The mean of the five results obtained shall be reported as adhesion to metal and self after immersion in water.

11. DETERMINATION OF WATER VAPOUR PERMEABILITY

11.0 Outline of the Method — Water vapour permeability is determined by applying the tape on the stainless steel box containing anhydrous calcium chloride and finding the increase in mass after 24 hours.

11.1 Number of Tests — Carry out the determination of three test specimens cut from the same roll.

11.2 Apparatus

11.2.1 *Stainless Steel Box* — with external dimensions approximately 9·5 × 2·5 × 2·0 cm closed completely except that the top has a centrally placed rectangular opening of 8 × 1 cm. The mass of the box shall be 25 g.

11.3 Procedure — Clamp one end of the tape in a support so that it hangs vertically and clamp a mass equivalent to 1 kg/cm width to the other end for about 5 minutes. Remove the tape from the clamp and apply it firmly over the opening of the box containing about 5 g of anhydrous calcium chloride. Trim off any overlap of the tape. Weigh the sealed box and place it in a desiccator containing water. Place the desiccator in an oven maintained at a temperature of 40 ± 1°C for 24 hours. Remove the box from the desiccator, allow it to cool and wipe off any adherent moisture with a clean dry cloth and weigh again.

NOTE — Care should be taken to see that the opening of the box is closed with tape as indicated, immediately after introducing the anhydrous calcium chloride in it.

11.4 Calculation

$$\text{Water vapour permeability, g/m}^2 = (M_2 - M_1) \times 1\,250$$

where

M_2 = mass in g after exposure, and

M_1 = mass in g before exposure.

11.5 Report — The mean of three determinations shall be reported as water vapour permeability in g/m^2 for 24 hours.

12. DETERMINATION OF DURABILITY

12.0 Outline of the Method — This test method defines conditions for determining the performance of adhesive bonds when subjected to continuous exposure at specified conditions of moisture and temperature. The performance is expressed as a percentage based on the ratio of strength retained after exposure to the original strength.

12.1 Apparatus

12.1.1 *Conditioning Cabinets or Ovens* — capable of maintaining a temperature of 70 ± 1°C, and humidity control.

12.1.2 *Stainless Steel Plates* — Same as in **5.2.2.**

12.2 Preparation of Test Plate — Clean the face of the stainless steel plate with a fresh piece of cotton wool or line rag saturated with sulphur-free toluene (*see* IS : 1839-1961*). Allow the cleaned plate to dry. Suspend the plate in the toluene vapour-bath for 5 minutes after the vapour line has reached the top of the plate so that no liquid is in contact with the plate. Allow the plate to cool for about 30 minutes at 27 ± 2°C and 65 ± 5 percent relative humidity prior to the test.

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*Specification for toluene, reagent grade.

12.3 Pre-Conditioning — Condition the roll of tape at 27 ± 2°C and 65 ± 5 percent relative humidity for 24 hours. Take out 60 cm of tape radially from the reel at a rate of 30 cm/min if the width of tape is 5 cm. If the width is more than 5 cm, remove the tape radially from the reel at the rate of 15 to 20 cm/min and cut to 5 cm width with sharp blade. The test piece shall not be allowed to touch any object prior to application.

12.4 Procedure

12.4.1 Place the prepared test plate, test surface upwards at the edge of the bench with one end nearer to the operator. Apply the test piece, without stretching, to the plate so that the test piece lies centrally on the plate and parallel to the longer side, with about 25 cm over hanging the end, taking care that no air bubbles are trapped between the tape and the plate. Cut off the excess tape. Allow the plate with applied test pieces to remain undisturbed for 5 to 6 minutes at 27 ± 2°C and 65 ± 5 percent relative humidity.

12.4.2 Suspend the plate with applied test piece for 7 days in a conditioning cabinet or oven in which the temperature is maintained at 70 ± 1°C and relative humidity 80 percent. At the end of this period, examine whether the tape remains firmly adhered to the surface of plate or not. Determine the adhesion strength as prescribed in **5.**

12.4.3 *Calculations*

Percent decrease in adhesion strength

$$= 100 - \left(\frac{A}{B} \times 100\right)$$

where

A = average strength when tested under the designated exposure conditions as given in **12.4.2**, and

B = original strength determined as prescribed in **5**.

12.4.4 *Reporting* — To pass the test, the tape shall remain firmly adhered to the surface of plate and the edges shall not show signs of lifting from position. The adhesion strength of the tape when determined under the designated exposure conditions shall not decrease by more than 12 percent of the original value.

13. DETERMINATION OF ADHESION TO SELF

13.0 Outline of the Method — Adhesion to self is determined by finding the force required to peel a strip of tape from its own backing at a specified angle and speed.

13.1 Procedure — Prepare the steel plate and apply the test specimen to it by the method prescribed in **5.4.1** and **5.4.2**, taking precautions not to touch the upper surface of the specimen. Turn the free ends over on to the back of the plate, removing the surplus. Apply the second test specimen to the backing of the tape already on the plate so that two strips are precisely coincident. Roll the second specimen with the roller and strip it by the method prescribed in **5.4.3**.

13.2 Calculation and Reporting — Calculate the load required to cause the separation of the tape from its own backing in terms of kilograms per centimetre width. If the variation of any of the five results exceeds ± 10 percent of the mean of the five test results, the results shall be discarded and a further set of five test specimens shall be tested. The mean of the five results obtained shall be reported as adhesion to self.

14. DETERMINATION OF STABILITY

14.0 Outline of the Method — Stability is determined by finding the adhesion strength of the tape after ageing at 50 ± 1°C for one week.

14.1 Apparatus

14.1.1 *Hot Air Oven* — Electrically operated, capable of being maintained at 50°C.

14.2 Procedure — Place the rolls of tape in an oven maintained at a temperature of 50 ± 1°C for a period of one week. At the end of this period, condition these rolls as prescribed in **4.1**. Determine the adhesion strength as prescribed in **5.**

14.3 Reporting — Report the percent decrease in adhesion strength, if any, due to ageing.